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STRUCTURAL CHANGES IN CHROMIUM DURING DEFORMATION

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STRUCTURAL CHANGES IN CHROMIUM DURING DEFORMATION[1]

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ABSTRACT: In recent years a number of detailed electron-microscopic studies have been published on the structural changes in metals with a body-centered cubic lattice, during deformation and recovery [1-5]. It has been established that the character of the structural changes in the deformation process depends essentially on the temperature and the speed of deformation. At relatively high temperatures (close to 0.3-0.5 T_{melt}) and relatively low speeds of deformation ($\varepsilon \approx 10^{-2}$-$10^{-5}$ sec^{-1}), an increase in the degree of deformation coincides with a development in the boundaries of a metallographic grain outside the so-called cellular structure. In this case the density of dislocations in the walls (boundaries) of the cells substantially exceeds the density of their distribution inside the cells. At fairly high deformation temperatures, the cells are practically free of dislocations. With a subsequent rise in the degree of deformation, the dimensions of the cells tend toward a certain minimum at a given temperature. It was also noted that the angle of disorientation of the neighboring cells increases with deformation [2-3], which conforms well with the increase in the density of dislocations in the cell walls.

At low temperatures (< 0.2 T_{melt}) and relatively high speeds of deformation, the density of dislocations inside the usual metallographically determined grain increases rather uniformly, and the cellular structure does not develop. There is no sharp boundary observed

[1] The materials of this paper were presented at the fifth All-Union Conference on Electron Microscopy in Sumy on July 7, 1965.

between these two extreme cases, and the cellular structure usually converts smoothly to a structure with homogeneous distribution of dislocations. An increase in the speed of deformation displaces the interval of transition into the range of higher temperatures [3].

Structural changes similar to the ones described above were also observed in deformation of crystals with dense packing and high packing-defect energy (for example, in Al, as in [4]*).*

In view of the high brittleness of metals with a body-centered cubic lattice, considering the essential structural sensitivity of the temperature in transition to a brittle state [6-8]*, a study of the change in the temperature of cold brittleness in connection with the structural changes accompanying preliminary deformation is of great interest. For the object of our study we chose chromium, as one of the metals which is most brittle at atmospheric temperature.*[2]

STUDY MATERIALS AND METHOD

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For our study material we took chromium which was purified of admixtures of elements with yttrium intrusion which (as was shown in [10]) has relatively high plasticity.

The chromium ingots were processed in two regimes (series A and B): extrusions on sheet bars and then, after intermediate annealing, they were laminated into bars of varying thickness. The material was further recrystallized and laminated at various temperatures (Tables 1 and 2).

In order to study the recovery processes, the chromium samples which were deformed by 20% at room temperature were kept in the furnace in a temperature range from 200 to 1200° C for one hour, and the samples which had passed through "thermal" lamination were kept in a range from 600 to 1200° C.

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[2] In preparing the manuscript for typing, we found a study [6] on the effect of dislocation distribution on the brittleness of chromium, several results of which were discussed in presenting the experimental material.

* Numbers in the margin indicate pagination in the foreign text.

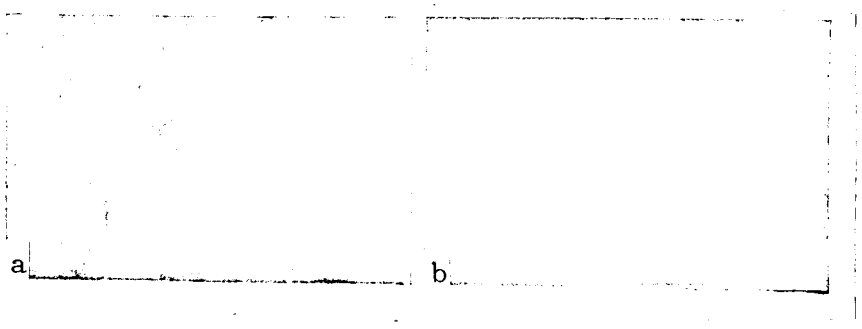

Fig. 1. Structures of Chromium Deformed at 20° C by 4% (a) and 20% (b), (×20,000). /113

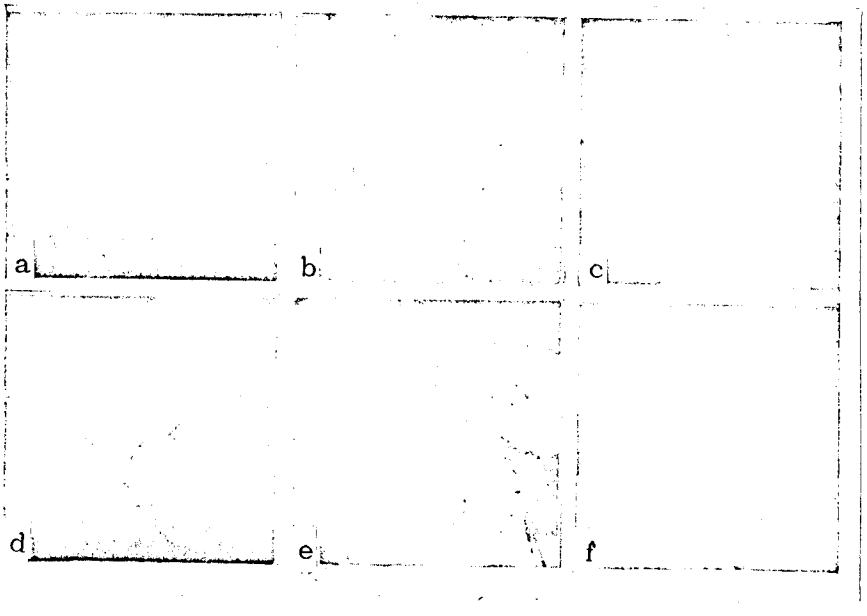

Fig. 2. Structures (×13,000), Observed During Annealing for One Hour, of Cold-Deformed Chromium at Temperatures of 200 (a), 400 (b), 600 (c), 800 (d), 1000 (e) and 1200° C (f).

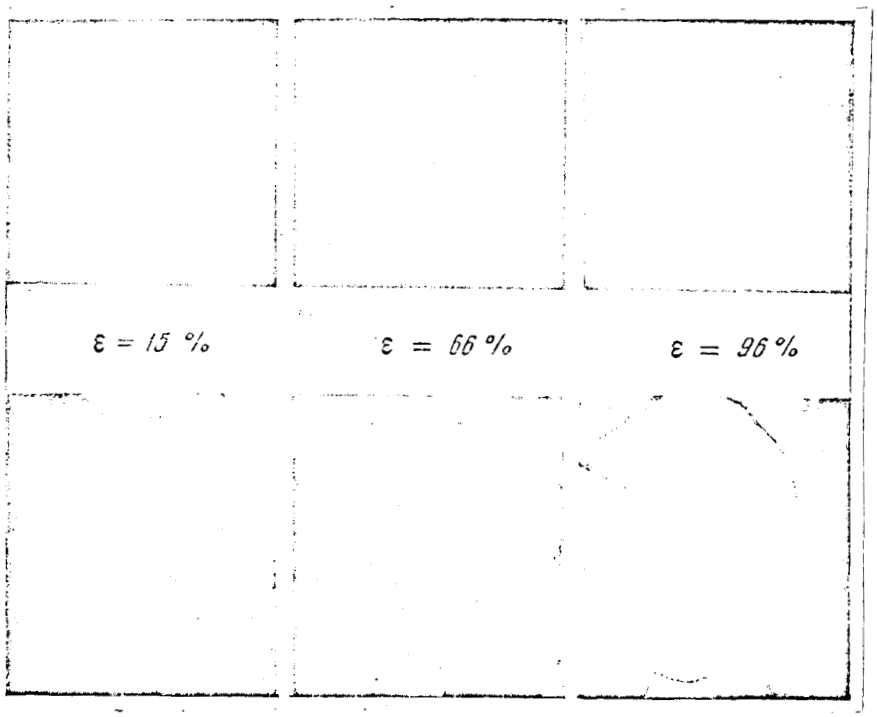

Fig. 3. Structural Changes in the Process of Thermal Lamination of Chromium at 550° C (×13,000). /114

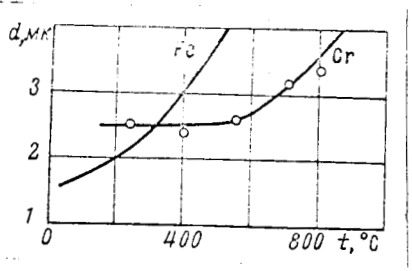

Fig. 4. Change in the Minimum Dimensions of the Cells Versus the Temperature of Deformation for Chromium and Iron. The Data on Fe were Given in [3].

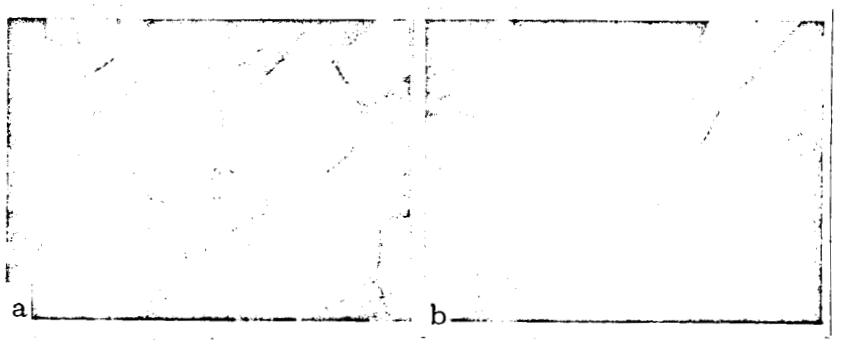

Fig. 5. Structural Changes Observed in Chromium Which Was Deformed at 550° C (ε = 95%) after Annealing for One Hour at 600 (a) and 800° C (b); (×10,000).

We prepared, for our electron-microscopic study, self-carrier samples of electropolishing in concentrated orthophosphoric acid. The bars for the self-carrier samples (circular discs with a diameter of 3 mm) were cut out of laminating plates by the electrospark method. The samples were studied "by transillumination" in an electron microscope UEMV-100 with an accelerating stress of 100 kV. /115

TABLE 1

Preliminary Processing	Processing Regime	
	Series A	Series B
Extrusion	1000° C	1200° C
Annealing in a vacuum of 10^{-5} mm Hg	1000° C - 1 hour	1200° C - 1 hour
Laminating to various Thicknesses	600° C	800° C
Annealing in a vacuum of 10^{-5} mm Hg	1000° C - 1 hour	1200° C - 1 hour

TABLE 2. LAMINATION REGIMES FOR THE CHROMIUM INGOTS

series A		series B	
t, °C	ε, %	t, °C	ε, %
20*	2.5, 4, 9, 20, 50, 72	800	31, 60, 86
240	60	800—600	90
550	15, 30, 66, 96	800—600—400	90
700	60	800—600—200	90

* The indicated degree of deformation was reached in one operation.

The cold brittleness experiments were conducted in samples with dimensions of 1 × 4 × 25 mm by bending at the velocity of permutation of the blade, 20 mm/min. After the temperature of transition from a plastic to a brittle state, we took that temperature at which the sample bent by 90° without fracturing, and below which it was fractured. The temperature range for the transition, as the experiments showed, was very narrow and did not exceed 10-15°.

STRUCTURAL CHANGES DURING DEFORMATION AND ANNEALING

The Structure of Chromium which is Deformed at 20° C. For low degrees of deformation, the dislocations have an irregular shape with a large number of jogs and kinks, which indicates the lightness of the cross-sectional slip and the high packing-defect energy. A significant quantity of dislocation loops is observed. With an increase in the degree of deformation, the dislocations combine into cloud-formation pile-ups, so that the dislocations are rather uniformly distributed throughout the volume of the sample.

The typical structures of chromium, deformed by 4 and 20%, are shown in Figure 1.

Structures During Annealing. The recovery begins at a temperature interval from 200 to 400° C. The dislocations which were uniformly distributed in the structure (Fig. 1) are reorganized in this case, forming a cellular structure (Fig. 2). With an increase in the annealing temperature, the dislocation density decreases, and the dislocations combine into regular networks, i.e., boundaries of torsion.

After 1200° C, the dislocations in the structure of the grain almost disappear.

Structure of Chromium Which Has Passed Through "Thermal" Lamination. In the process of thermal lamination, the superposition of the simultaneously occurring processes of hardening and softening results in the formation of a clearly observed cellular structure (Fig. 3). /116

Even at the earlier stages of deformation, cells are formed whose measurements vary rather weakly in the course of further plastic deformation; in this case, the density of deformations in the walls of the cells increases, which coincides with an increase in the angle of disorientation among the neighboring fragments, reaching 15-20° during the final stages of deformation.

We found the relationship of the dimensions of the deformation cells to the lamination temperature (Fig. 4). Moreover, the disorientation among the neighboring cells in the case of lamination at 800° C is substantially lower than at 550° C; this is probably due to the fact that, at a higher deformation temperature, the portions of annihilating dislocations, which do not take part in the formation of a cellular structure, increase.

As the degree of deformation increases, the walls of the cells lose their clearly expressed dislocation structure and acquire a typical type of grain boundary.

Structures for Annealing after "Thermal" Lamination. The deformations substructure, obtained by thermal lamination, is not preserved with subsequent heating.

We can see from Figure 5 that during heating the cells behave like grains during collective recrystallization - separate fragments increase intensely because of the neighboring ones.

DISCUSSION OF THE RESULTS

The formation of a cellular structure during the process of deformation is accompanied by an increase in the angle of disorientation for the neighboring fragments. Disorientation very rarely

increases when the dimensions of the cells approach a minimum value, since the density of dislocations in the boundaries continues to increase with an increase in the degree of deformation. With fairly high deformation ($\varepsilon \approx 80$-90%), the maximum angle of disorientation exceeds 15-20°.

Such strongly disoriented boundaries should actually be the regular grain boundaries. A transfer of plastic deformation through such boundaries should be accompanied by the formation of an accumulation of dislocations, and the length of the slip plane of the accumulation, which coincides at earlier stages of plastic deformation with measurements of a metallographically detected grain with a clearly defined structure in the sample, will be limited by the measurements of an individual cell (or fragment).

Thus, an increase in the degree of plastic deformation qualitatively changes the character of the cell boundary, as impediments to dislocation movement. If with small values of ε (and thus with small disorientations) the cell boundary shows resistance to dislocation movement by a type of resistance of "forest" dislocations, then for disorientations higher than 5-6° (we will agree to call this disorientation critical), the boundaries of the cells fulfill the functions of grain boundaries. Distribution of the number of boundaries by the degree of their disorientation changes with an increase in the degree of deformation towards an increase in the number of strongly disoriented boundaries.

The observed structural changes which accompany the thermal lamination process must be substantially expressed in the mechanical properties of the metal. The regular metallographically defined grain is extended with an increase in the degree of deformation in the direction of lamination, and its diameter in the plane which is perpendicular to the lamination decreases. However, this appearance of a disoriented cellular structure, whose formation is actually equivalent to the size reduction of a grain, at least by one order of magnitude, must play a significant part in the change in the mechanical properties. /117

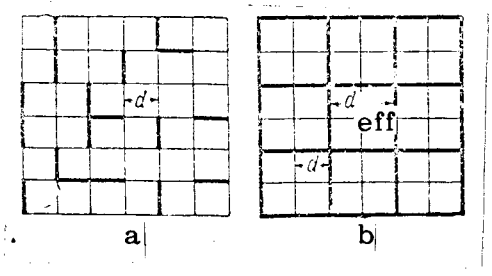

Fig. 6. Diagrams of Possible Arrangements of Strongly Disoriented Boundaries (Indicated by the Heavy Lines).

Considering that the deformation is accompanied by an increase in the number of boundaries of the cells which have disorientation higher than critical, we can draw a diagram illustrating a continuous change of a certain effective measurement for the substructural formations d_{eff}, which form the basic mechanical properties of the metal.

Two possible variations for the arrangement of strongly disoriented boundaries of the cellular structure are shown in Figure 6. Considering the

minimum elastic energy, the second arrangement (Fig. 6b) seems more probable. It is obvious that, with an increase in the degree of deformation, the value for d_{eff} will decrease continuously, approaching the measurements of the cell d, while the dimensions of the unit cell, d, actually depend very little on the degree of deformation (Fig. 7).

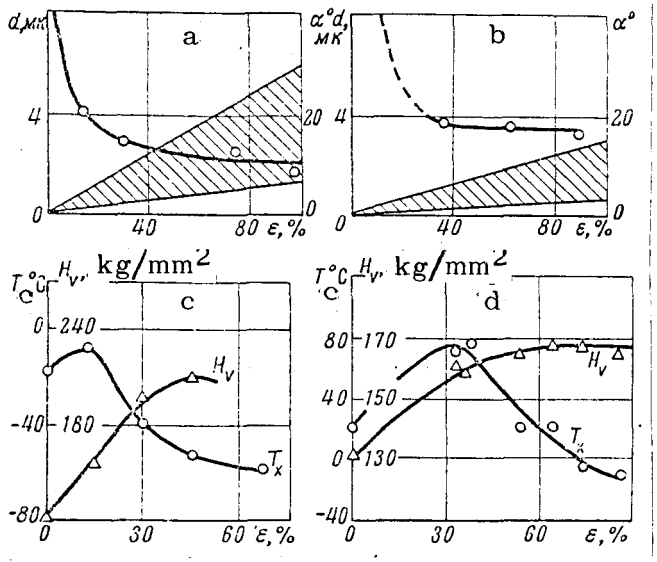

Fig. 7. Changes in the Structure (a,b) and Properties (c,d) during Lamination of Chromium at 550 (a, c) and 800° C (b,d).

A gradual increase in the hardness of the metal can be observed experimentally with an increase in the degree of deformation. As long as the hardness is a characteristic roughly proportional to the yield point and linked with the resistance of the material to plastic deformation, we can use, in first approx- /118 imation for a qualitative analysis of the change in hardness, the relationships which link the yield point with the grain sizes. According to Stroh and Petch [6,11], the yield point σ_s is linked with the grain size d by the relationship

$$\sigma_s = \sigma_0 + Kd^{-\frac{1}{2}} \qquad (1)$$

where σ_0 is the resistance to dislocation movement, and K is the parameter which characterizes the process of the slip transfer through the grain boundary [11]. In measuring the hardness, the dimensions of the impression covered in area a large number of the cells of the substructure, and thus the value for the hardness was undoubtedly determined by the processes of extending the deformations to a significant number of cells with a cross-section of the substructural boundaries. As d_{eff} and the transition of an increasing number of boundaries into the category of grain boundaries decrease, the hardness of the metal increases, revealing a tendency towards saturation, since with fairly high degrees of deformation a large portion of the boundaries has disorientation which exceeds the critical, and $d_{eff} = d$.

The value of σ_0, obviously, does not change significantly with an increase in the degree of high-temperature deformation, since the range of the lattice inside the cells is practically free of dislocations even with high overall pressing. Moreover, a possible development of the processes of deformation aging at high laminating temperatures (500-700° C), and a decrease in the concentration of elements imbedded in the die connected with this aging, are cap-

able of decreasing somewhat the amount of resistance to dislocation movement.

Deformation aging can occur in chromium relative to its low purity. While special studies [10] on the high-purity chromium doped with yttrium did not reveal any signs of deformation (and hardening) aging, it can be observed, for example, in chromium with 0.005% N_2, in which neither yttrium nor other highly active elements are introduced.

The high sensitivity to formation of a disoriented cellular structure should also be detected by the temperature of the cold brittleness T_{cb}.

$$T_{cb}=\frac{\sigma_{0s}-{}^1/_4 Kl^{-1/2}}{\frac{R}{V}\ln\left[\frac{N}{\varepsilon}\left(\frac{l}{d}\right)^{1/2}\right]} \qquad (2)$$

where σ_{0s} is the resistance to dislocation movement at absolute zero; d is the size of the grain; l is the size of the mosaic blocks; V is the activation volume; K is the constant of the Mott-Stroh equation for the real value of the destroying stress $\sigma_{hv} \approx \sigma_0+Kd^{-\frac{1}{2}}$; $\dot{\varepsilon}$ is the speed of deformation; R is the Boltzmann constant; and N is the parameter.

The sharp decrease in d from the values of the diameter of the metallographic grain for small degrees of lamination deformation to values of the diameter of strongly disoriented cells must coincide with a decrease in T_{cd}, which is also observed in experiment (Fig. 7). The state of the cold brittleness temperature, at each determined degree of plastic deformation after formation of a cellular structure, is determined actually by the value of d_{eff}, which should be put into (2) in the place of the value for d. A change in T_{cd} at t_{def} = 550° C occurs in absolute correspondence with the law predicted in (2) (Fig. 7a,c).

In the high-temperature range of deformation (0.2 $T_{melt} < T_{def} < T_{recr}$) at a fairly high degree, the formation of mosaic blocks actually does not occur; there is only a cellular structure as the final result of deformation. Even after deformation which exceeds $\varepsilon \approx 90\%$, there is no "forest" dislocation, and on the x-ray photographs the lines are broadened and widened. /119

The structural changes at high-temperature deformation (which, however, occur below the recrystallization temperature) are extremely unusual; despite such desirably large deformation, the material remains unhardened, since the dislocation density inside the cells practically does not increase in comparison with the original state. An important result of such deformation is the formation of a new, exceptionally small grain (on the base of the strongly disoriented cellular structure). The reinforcement observed in this case is

related, basically, to a decrease in d_{eff}, and not to a change in the density of "forest" dislocations.

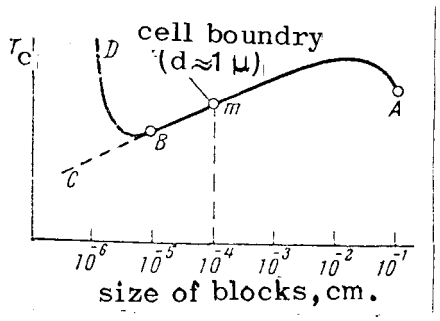

Fig. 8. Diagram of the Temperature of Cold Brittleness Versus Size of the Blocks; Point *m* corresponds to the Size of the Blocks Which Coincide with the Size of the Cells.

In the range of low-temperature deformation ($T_{def} \leq 0.2\ T_{melt}$), as we noted earlier, no cellular structure is formed, and the hardening results mainly in an increase in the density of "forest" dislocations. In this case, naturally, there is "crushing" of the mosaic blocks, and, as a result, significant hardening of the x-ray diffraction lines. In the intermediate temperature range, where superposition of these two structural states is observed, the hardening is related to the combined effect of the disoriented cells and blocks. In [12], where the influence of the measurements in blocks of a mosaic l on the cold brittleness temperature T_{cb} was first presented, this work indicated the complex character of the relationship $T_{cb} = f(l)$ (Fig. 8).

However, curve *ABC* (Fig. 8) obtained in [12] did not take into account the changes in the value of σ_{0s} during deformation. For metals with a body-centered cubic lattice, as we know from [7], the value of σ_{0s} is mainly determined by three components:

$$\sigma_{0s} = \sigma_{PN} + \Sigma\sigma_i + \sigma_f \qquad (3)$$

where σ_{PN} is the resistance of the lattice to dislocation movement (Peierls-Nabarro stress); $\Sigma\sigma_i$ is the sum of resistances related to the different types of interaction of the point defects (atoms with interstitial impurities, vacancies, etc.) with the field of dislocation stresses; and σ_f is the resistance of "forest" dislocations.

Despite the fact that metals in the VI *A* group at temperatures for transfer to brittle fracture have a particularly large "specific weight" for the first two components, and we can therefore consider that *ABC* is a good approximation for chromium, the value for σ_f for a strongly hardened metal must increase significantly. This results in the fact that $T_{cb} = f(l)$ must follow the law for *ABD* (Fig. 8). The possibility for reaching a peak on curve *ABC* (or *ABD*) depends essentially on the value for the original grain [12]. The position corresponding to the formation of a cellular structure ($d_{eff} = d$) is roughly plotted in Figure 9 (since we cannot have $l > d$).

It follows from this that the additional low-temperature deformation of a metal, in which a cellular structure is formed by preliminary deformation at high temperatures, can be accompanied by a decrease in T_{cb} if the dimensions of the mosaic blocks do not

go beyond the limits of the *mB* section. We actually succeeded in observing such a position for T_{cb} (Fig. 9). The chromium, deformed /120 at 800 and 600° C (i.e., in the temperature range for formation of a cellular structure), was also laminated (with 4% reduction at 400 and 20° C). In this case the appearance of dislocations inside the cells was observed in an electron microscope, and the transition temperature decreased to a greater degree (Fig. 9).

From the point of view of the relationships examined, the deformation which has been conducted completely in the low-temperature range is obviously less preferable for a decrease in T_{cb}, since the dimensions of the grain in this case remain equal to the dimensions of metallographically defined grains, and the high density of dislocations by cold hardening can cause entrance into the *BD* section. Actually, lamination of chromium at 20° C by 47% (in one operation), although it did not cause fracture of the sample in the laminating process, did increase, however, the value for T_{cb} practically to the range of room temperatures.

The effect of an increase in T_{cb} at earlier stages of deformation at high temperatures is also related to the unusual appearance of the effect of a mosaic structure.

At the first stages of cellular structure formation, when the angle of disorientation for the cells is less than critical, we can

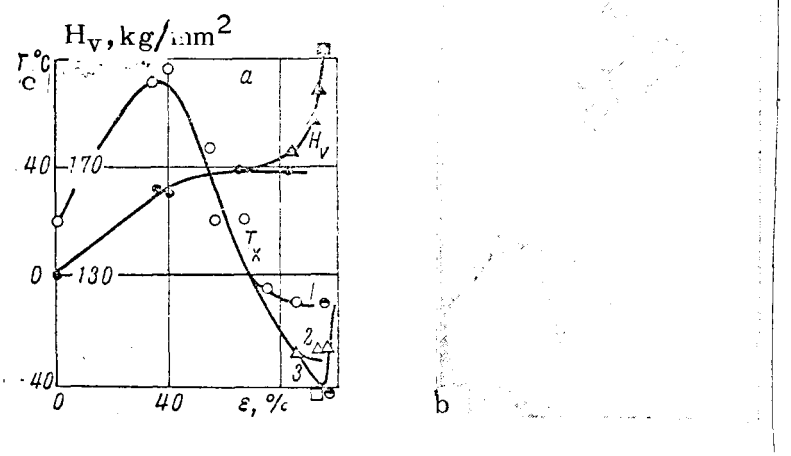

Fig. 9. (a) Change in the Cold Brittleness Temperature and in the Hardness Temperature Versus the Degree of Deformation: ● ○ is the Deformation at 800° C (1); Δ ▲ is the Additional Deformation at 600° C (2); ■ ⊏ is the Additional Deformation at 400° C (ε = 12%); ◖ is the Additional Deformation at 20° C (ε = 4.12%): (b) Photograph "by Transillumination" of Chromium Which has Undergone Deformation at 800, 600, and 20° C (×20,000).

examine them as large mosaic blocks. In this case d_{eff} coincides with the dimensions of a metallographically defined grain, and if it is sufficiently large ($d \approx 10^{-1}$ cm, see Fig. 8), then T_{cb} can change such as would follow from Figure 7d.

In the series of experiments conducted we established the fact that the value of the rise in T_{cb}, with a rise in the degree of high-temperature (at the first stages) deformation, unconditionally reveals the dependence on the dimensions of the original (before lamination) grain. Thus, for example, the small-grain samples laminated at 550° C (original measurements of the grain were d = 10-15μ) practically did not show a maximum (Fig. 7a,c), and in large-grain /121
samples which were deformed by lamination at 800° C (original dimensions of the grain were $d = 10^{-2}$-10^{-1} cm, Fig. 7b,d) it was very clearly observed. Lamination at a higher temperature in the second case enabled us to find this effect, since (as the electron diffraction patterns showed) the angle of disorientation at 800° C increased much more slowly (Fig. 7b).

In a paper we already mentioned [9], Garrod and Wain drew the conclusion that the critical condition for brittle fracture of chromium is not a development, but a nucleation of the crack. This conclusion corresponds with our assumptions [12] and lays the foundation for attempting to write (2) (which we did in our paper) for analyzing the conditions for transition from plastic to brittle fracture, since this equation was obtained for critical conditions of nucleation of brittle cracks (see in relation to this the discussion in [14] and [15]).

The structural changes studied in this paper, which coincide with the processes of relaxation, polygonization and recrystallization of deformed chromium, are in correspondence with well-known experimental data on the change in T_{cb} and several other properties with similar developments. Thus, for example, the change in T_{cb} and H_V in deformed chromium (whose chemical composition and method for obtaining it were similar to the ones followed in our paper), after annealing at various temperatures, was studied in [10]. A significant change in T_{cb}, as we would anticipate, was observed after annealing (for 2 hours) at 600-800° C, when the fracture of the cellular structure and the intense increase in individual cells begin according to the type of process involved in the collective recrystallization.

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